Substrate-dependent electronic structure of an organic heterojunction

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This work focuses on organic-organic heterojunctions (OOHs) formed between two small-molecule, low band-gap, semiconductors, tris{2,5-bis(3,5-bis-trifluoromethyl-phenyl)-thieno}[3,4-*b*,*h*,*n*]-1,4,5,8,9,12-hexaaza-triphenylene (THAP), and copper phthalocyanine (CuPc). The organic layers are deposited on various substrates, and the energy level alignment between them is investigated by ultraviolet photoemission spectroscopy. The electronic structure of the OOH is found to depend on the work function of the organic underlayer predeposited on the different substrates. The vacuum level offset between THAP and CuPc, which consists of the sum of the interface dipole and the molecular level shift, varies from 0.26 to 1.37 eV. The interface dipole between the two organic films linearly changes with the work function of the organic underlayer.

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INTRODUCTION

Organic-organic heterojunctions (OOHs) play a central role in multilayer organic devices, such as light-emitting diodes and photovoltaic cells. Molecular level offsets across interfaces control charge transport or separation[1] and device performance. Therefore, understanding the mechanisms that dominate the electronic structure of OOH is important for device design and engineering.

Vacuum level alignment has been observed across many OOHs, a result that has been attributed to two main factors: (i) weak interfacial interaction at interfaces between van der Waals (vdW)-bonded molecular materials and (ii) lack of free carriers in solids with relatively large highest occupied molecular orbital-lowest unoccupied molecular orbital (HOMO-LUMO) gaps. Interfacial charge displacement and potential shift are minimized, leading to a straightforward relationship between molecular level energies across the interface. Yet, vacuum level shifts and interfacial dipoles on the order of a few hundred millivolts have also been reported for some OOHs,[2,3] and these cases have raised questions regarding the overall mechanisms that control the electronic structure of these interfaces.[4]

The electronic structure of an OOH is, in principle, uniquely defined by the properties of the two constituent materials, namely, their energy gap, electron affinity (EA), and ionization energy (IE). In that sense, the electronic structure of the OOH is expected to be independent of the electronic properties of the substrate on which the two organic layers are deposited, in particular, of its work function (WF), as long as the substrate does not significantly alter either the physical nature of or the charge density in the bottom organic layer. The structure of the OOH is also expected to be independent of the sequence of deposition of the two organic materials, i.e., A on B vs B on A, as a consequence of interfacial vdW interactions, in contrast to the defect—or dangling bond—dominated interfaces generally formed by epitaxy between covalent bonded inorganic solids. Under such conditions, experiments have indeed shown OOH energetics uniquely defined by the constituents, and the model based on induced density of interface states (IDIS) and alignment of charge neutrality levels (CNLs) has been shown to relatively explain well both molecular level offset and interface dipole at OOHs.

As an exception to the above rule, Tang *et al.*[5,6] recently reported a significant substrate-induced effect on the vacuum level offset at the heterojunction between copper-phthalocyanine (CuPc), and tris(8-hydroquinolinato) aluminum (Alq_3). Using ultraviolet and x-ray photoemission spectroscopies (UPS and XPS), they showed the occurrence of an interface "band bending" and the formation of a space-charge region that compensate for the shift of the Fermi level (E_F) caused by a change from high to low WF substrate. Doping one of the two organic layers, which leads to a shift of E_F in the materials, has also been reported to modify the molecular level offsets at an OOH.[7] In the present paper, we focus on the energetics of an OOH formed between two small-gap molecular solids, CuPc and tris{2,5-bis(3,5-bis-trifluoromethyl-phenyl)-thieno}[3,4-*b*,*h*,*n*]-1,4,5,8,9,12-hexaazatriphenylene (THAP) (Fig. 1). CuPc is a well-known hole-transport material with an energy gap of 1.9 eV and IE=5.2 eV.[8] THAP is a high mobility electron transport material with an energy gap of 1.8 eV and IE=6.4 eV.[9] These materials make an interesting donor-acceptor pair for photovoltaic applications. In our work, the OOH is built on substrates with varying WF in order to tune the position of E_F in the band gap and to investigate subsequent electronic effects.

EXPERIMENT

CuPc was purchased from Sigma-Aldrich and purified twice by zone sublimation methods. THAP was synthesized and purified using column chromatography as previously described.[10] Both compounds were placed in thermal evaporation cells mounted on the ultrahigh vacuum (UHV) growth chamber (base pressure $\sim 5 \times 10^{-10}$ Torr) of a multichamber surface analysis system, and thoroughly degassed prior use.

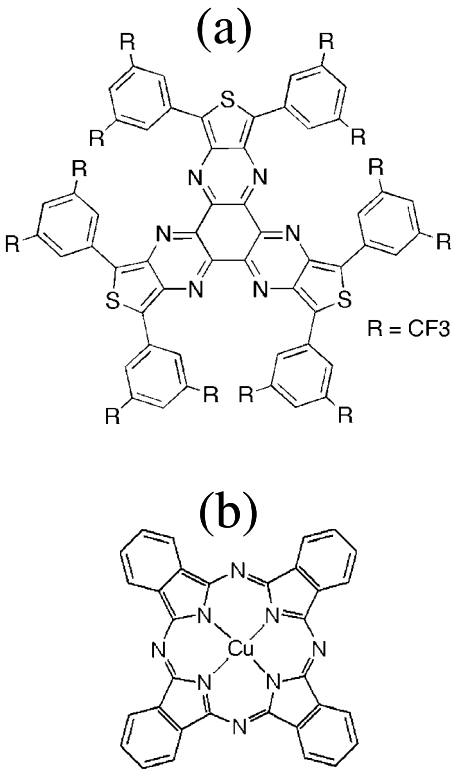

FIG. 1. Chemical structure of (a) THAP and (b) CuPc.

Sublimation temperatures were 390 and 350 °C for CuPc and THAP, respectively. Deposition rates (~0.5 Å/s for bottom layer, 0.1 Å/s for overlayers) and film thickness were monitored via quartz microbalance.

Substrates with different WFs were prepared in order to move E_F in the band gap of the two molecular films. Gold substrates [Au(1500 Å)/Ti(500 Å)/Si] were (i) cleaned with acetone and methanol without further treatment; (ii) treated *ex situ* by UV ozone for an hour; (iii) sputter cleaned *in situ* with 1 keV Ar^+. Another substrate used was poly-3,4-ethylenedioxythiophene-polystyrenesulfonate (PEDOT:PSS) CH8000 from Baytron and was spun coated onto Indium tin oxide (ITO) (the ITO was precleaned by standard procedures, including ultrasonic bath in detergent, solvents, and UV ozone for 1 h). The PEDOT:PSS was annealed at 180 °C in a nitrogen atmosphere and further annealed in vacuum at 180 °C for 20 min. Finally, aluminum films covered with a native oxide (AlO_x) were rinsed with solvent and blown dry with nitrogen.

Once the substrates were introduced in the UHV chamber, all further treatments, organic film growth, and UPS and XPS analyses were carried out without breaking vacuum. Work function and valence state measurements were performed by UPS, using a helium discharge lamp (He I, $h\nu$ =21.22 eV). The WF was determined using the position of the Fermi level of the system and the vacuum level (E_{vac}) deduced from the cutoff of photoemission.[11] Similarly, the IE of each organic film was determined using E_{vac} and the onset of the highest kinetic energy feature in the UPS spectrum.[11] The CuPc and THAP EA were determined separately using inverse photoemission spectroscopy.[8,9] In each of the experiments described below, the combined thickness of the bilayer molecular films (CuPc+THAP) was kept below 80–100 Å to prevent charging during UPS. The resolution of the UPS measurement was 0.15 eV, as defined by the width of the Fermi step measured for a sputtered polycrystalline Au film. Chemistry at interfaces was monitored via XPS (Al $K\alpha$ line, 1486.6 eV). The overall energy resolution in XPS was 0.9 eV.

RESULTS AND DISCUSSION

The WF of the substrates and the IE and EA of the two organic materials are summarized in Table I. The PEDOT:PSS WF is about 0.5 eV higher than usually reported,[12] but is in good agreement with the value reported by Koch *et al.* for films annealed in vacuum.[13] These various substrates lead to a range of Fermi level positions in the gap of the bottom organic film, as will be seen below.

Small molecules adsorbed onto atomically clean Au are generally found to lower the metal WF, leading to an interface vacuum level shiftdown from the metal. This results in a larger hole-injection barrier than might be expected from the initial values of the WF of clean Au and the IE of the organic material. The same molecules adsorbed onto a nonmetallic electrode with WF similar to that of Au, such as PEDOT:PSS, form an interface with smaller vacuum level shift and smaller hole-injection barrier, as has been demonstrated with pentacene and α-NPD.[14,15] We note at the outset, however, that THAP deposited on sputter-cleaned Au (WF=5.43 eV in the present case) reduces the electrode WF by 0.1 eV or less. Similar observations have previously made with molecules including perfluorinated copper phthalocyanine (F_{16}-CuPc)[16] and perylenetetracarboxylicdianhydride (PTCDA)[17] which, like THAP, have large EAs. In these cases, a large downward movement of molecular levels of the organic films is prohibited because electrode Fermi level E_F cannot be overlap with the large unoccupied states tailed from the LUMO into the band gap.

The first OOH to be discussed consists of a CuPc film with thickness ranging from 2 to 16 Å incrementally deposited onto a 50 Å THAP film, itself predeposited on an as-

TABLE I. Work function of the various substrates used in this work: native aluminum oxide (AlO_x), contaminated Au (*c*Au), UV ozone-treated Au (*o*Au), sputtered Au (*s*Au), PEDOT:PSS; IE/EA of the organic thin films: CuPc and THAP

	AlO_x	*c*Au	*o*Au	*s*Au	PEDOT:PSS	CuPc	THAP
WF (eV)	3.65	4.75	5.38	5.43	5.65		
IE/EA (eV)						5.04/3.1	6.41/4.6

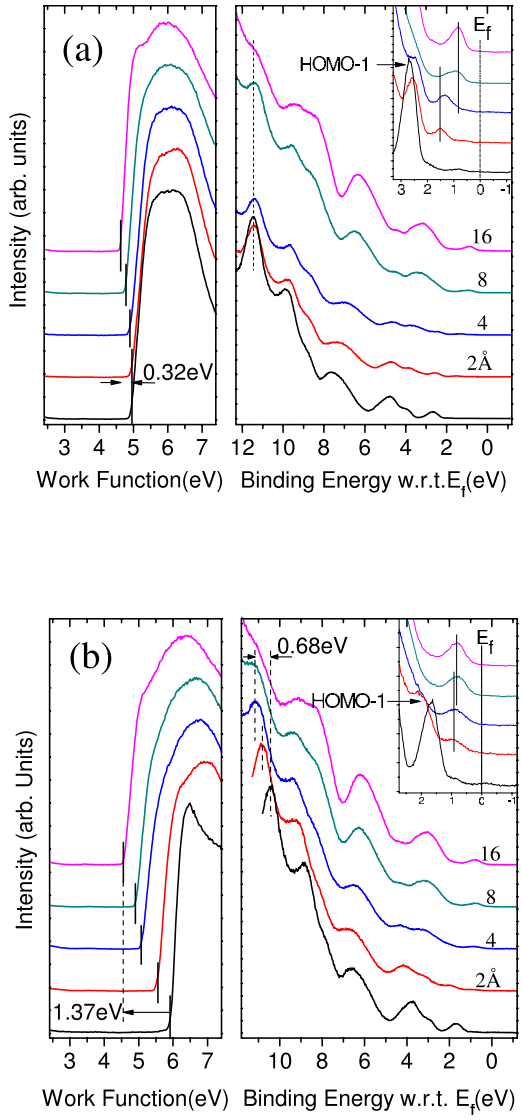

FIG. 2. (Color online) UPS spectra of CuPc incrementally grown on 50 Å THAP with (a) contaminated Au (cAu) and (b) PEDOT:PSS as substrate. The vertical lines on the left panel indicate the work function at each coverage, as well as the amount of total vacuum level offset ΔE_{vac}. The dashed lines on the right panels outline the position of a specific THAP feature as a function of CuPc coverage. Insets: magnified valence structure close to E_F. Two vertical lines point out the position of new feature and CuPc HOMO.

loaded Au substrate (cAu, WF=4.75 eV). The corresponding UPS valence spectra are shown on the right of Fig. 2(a). The binding energy of the valence spectra is taken E_F as reference. The secondary electron cutoffs are displayed on the left panel of Fig. 2(a) on an energy scale that gives the WF of the film. The IE of the bare THAP film is 6.41 eV, in good agreement with a previously reported value.[9] The THAP UPS features are almost entirely quenched with a nominal CuPc thickness of 16 Å, indicating that the latter material effectively wets the THAP underlayer. The subsequent spectra (for CuPc thicknesses >16 Å, not shown here) are dominated by photoelectrons from CuPc. The CuPc IE is 5.04 eV, also in very good agreement with published data.[8] Upon deposition of CuPc, the WF of the film decreases by a total of 0.32 eV with respect to that of THAP/cAu 4.94 eV [left of Fig. 2(a)], although the energy position of the THAP features remains unchanged, as seen from the 11.4 eV peak. This indicates that CuPc does not induce any significant "band bending" in the original THAP layer. Most of the decrease in WF occurs at CuPc thicknesses between 8 and 16 Å, when the CuPc HOMO becomes clearly identifiable. The inset of Fig. 2(a) shows that a new feature located at 1.5 eV below E_F appears at intermediate coverage, and disappears from the CuPc spectrum at 8 and 16 Å. This interface state, localized at the THAP/CuPc interface, will be discussed below. Note that the small features appearing at, and even above, the Fermi level in the insets of Figs. 2(a) and 2(b) are replicas of the HOMO-1 peak due to the presence of the weak parasitic 23.09 eV He radiation line.

The second OOH was formed with the same deposition sequence and film thickness, but on a PEDOT:PSS substrate annealed in vacuum (WF=5.65 eV). The initial THAP(50 Å)/PEDOT:PSS layer has a WF equal to 5.94 eV, and the HOMO onset is at 0.5 eV below E_F [Fig. 2(b)]. In contrast to the previous case, the deposition of the smallest amount of CuPc (2 Å) causes a significant downward shift of the vacuum level, namely, a decrease of the film WF and a similar shift of the THAP valence features toward higher binding energy [e.g., the 11 eV peak in Fig. 2(b)]. The total shifts with complete CuPc coverage are 1.37 and 0.68 eV, respectively. As in the previous case, an interface state appears at low CuPc coverage [~0.98 eV in the inset of Fig. 2(b)], although its position does not allow a clear distinction from the overlayer HOMO.

XPS shows that the THAP F1s, S2p, and C1s core levels shift by 0.55, 0.46, and 0.41 eV toward higher binding energy, respectively, for the intermediate 4 Å coverage of CuPc. These shifts are slightly smaller than those observed in UPS, but still in agreement given the XPS resolution. No further shifts are seen with increasing CuPc coverage. The Cu2p peak energy appears to be independent of the CuPc coverage.

Additional OOHs were formed on sputtered gold (sAu, WF=5.43 eV) and UV ozone treated gold (oAu, WF =5.38 eV), which led to intermediate Fermi level positions in the gap of the THAP underlayer, and, thus, to intermediate WFs equal to 5.4 and 5.75 eV, respectively. As a side note, the fact that the WF of the organic layer does not monotonically increase with the electrode WF shows again that the interface position of E_F in the gap of organic films reflects more than the initial substrate WF, and depends on the nature of the organic/substrate interaction.[18] Interaction of THAP molecules with the atomically clean metal surface (sAu) is clearly different from the electrode modified by contamination (cAu) or product of UV ozone cleaning (oAu), and with the nonmetallic electrode (PEDOT:PSS).

The interface energetics deduced from these experiments are summarized on the left of Fig. 3 (CuPc on THAP), where four energy diagrams are arranged in ascending order of initial THAP WF. Note that the IEs measured on both CuPc and THAP films are constant within experimental error in the experiments under discussion, indicating that the different pretreatments of the substrates do not significantly affect the

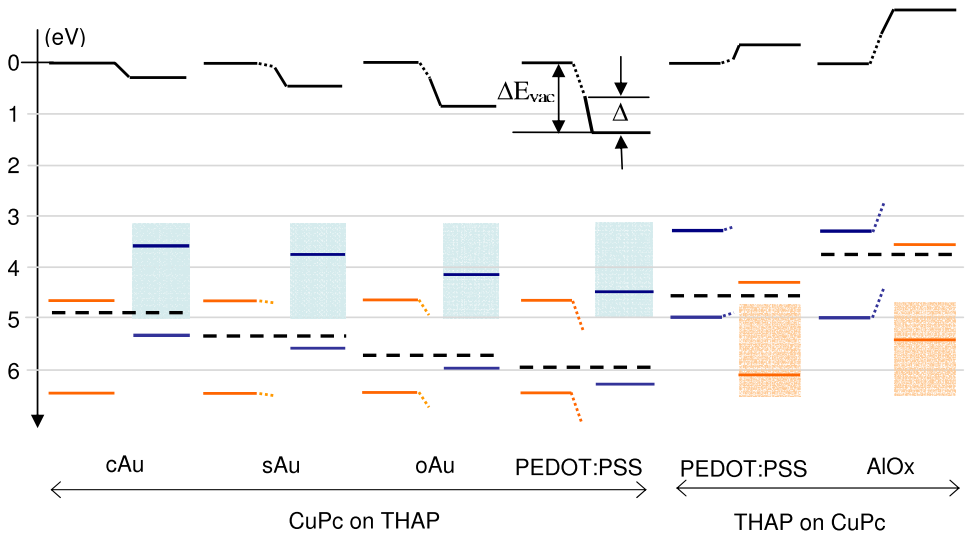

FIG. 3. (Color online) Energy diagrams of CuPc/THAP and THAP/CuPc heterojunctions on various substrates. CuPc and THAP HOMO and LUMO are shown in blue (dark gray) and orange (gray), respectively. The CuPc and THAP LUMO positions are obtained by subtracting the transport gap [1.75 eV for THAP (Ref. 9) and 1.9 eV for CuPc (Ref. 8)] from the material IE. The dashed horizontal lines indicate the position of E_F. The curved dotted lines represent the shift of molecular energy levels upon deposition of overlayers. Semitransparent blue (light gray) and orange (gray) blocks guide the eye to the position of the CuPc and THAP transport gaps, assuming vacuum level alignment.

electronic properties of the organic thin films. As the THAP WF approaches, and then exceeds, the value of the CuPc IE, which is the case with *s*Au, *o*Au, and PEDOT:PSS, electrons are transferred from the CuPc HOMO to the THAP. Equilibrium is reestablished when the CuPc molecular levels move downward to keep the HOMO nearly filled below E_F. The charge transfer increases the interface dipole and the band bending in the THAP layer. It is difficult to assess whether the charges are uniformly distributed in the THAP film, as in a space-charge region, or localized on few molecular layers next to the interface. Thus, the total shift is simply represented by dotted lines in Fig. 3. As a side note, we cannot rule out the possibility that some pinholes are present in the bottom organic layer. However, the shift of molecular levels described above is strong evidence of direct charge exchange between the two organic constituents, and the overwhelming majority of the interface area probed in our experiment corresponds to the organic-organic interface.

The total vacuum level offset across the interface, ΔE_{vac} (Fig. 3), is the shift from the vacuum level of the pristine THAP layer to that of the fully grown overlayer. The difference between ΔE_{vac} and the molecular level shift in THAP (measured using the 11 eV feature and the THAP core levels) is the interface dipole Δ (Fig. 3) at the CuPc/THAP heterojunction. Both ΔE_{vac} and Δ are plotted as a function of the initial THAP WF in Fig. 4. Interestingly, Δ linearly varies while the molecular level shift and ΔE_{vac} increase superlinearly. This demonstrates that E_F is not pinned at a given position in the CuPc gap, but moves as a function of substrate WF. If E_F was fixed, ΔE_{vac} would also linearly vary with the WF of THAP. As E_F moves down with different substrates, the CuPc HOMO is pushed down as well. The amount of charge transferred to THAP depends on how deep E_F penetrates into the CuPc density of states extending from the HOMO into the band gap. This is presumably the origin of the nonlinearity of the charge transfer, and, thus, of the total vacuum level offset, as a function of the THAP WF.

The electronic structure of heterojunctions formed by the reverse deposition sequence, i.e., THAP on CuPc, was studied for both high and low WF substrates: PEDOT:PSS (5.65 eV) and AlO_x (3.65 eV). The results are displayed on the right part of Fig. 3. For CuPc/PEDOT:PSS, E_F is 0.3 eV above the CuPc HOMO edge and the WF of the initial CuPc film is 4.53 eV, slightly less than the EA of THAP. As the overlayer is deposited, some charges are transferred from CuPc to raise the THAP LUMO edge above E_F and bring the system into equilibrium. The WF increases by 0.32 eV while the molecular levels of the underlying CuPc barely shift toward lower binding energy (0.06 eV), resulting in a 0.26 eV

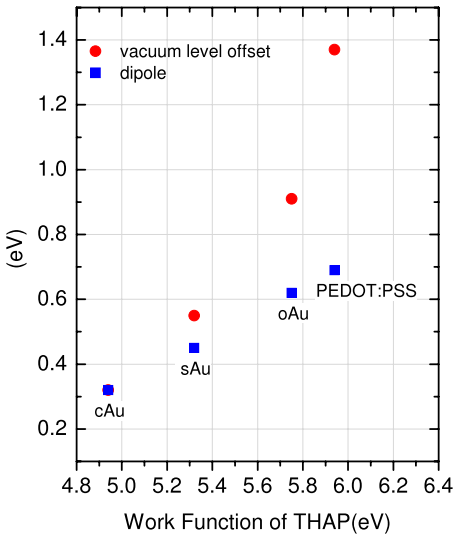

FIG. 4. (Color online) Vacuum level offsets (●) and dipole (■) at the CuPc(16 Å)/THAP interface as a function of initial THAP WF. The difference between the two represents the shift of underlying THAP molecular levels.

interface dipole. No interface state is observed in this case.

In the AlO_x case, E_F starts high in the CuPc gap, giving a low WF of 3.73 eV, which is ~0.9 eV smaller than the EA of THAP. Thus, a substantial electron transfer occurs from CuPc to THAP, which raises its LUMO and shifts the CuPc molecular levels upward to reestablish equilibrium. Subtracting the 0.6 eV molecular level shifts from the overall ΔE_{vac} of 1.11 eV leads to an interface dipole Δ=0.51 eV.

The interface state mentioned above (insets of Fig. 2) is observed at all the CuPc-on-THAP interfaces, regardless of the substrate. Chemical reaction does not appear to be at the origin of this state, based on the lack of evidence from XPS. Its binding energy (with respect to E_F) varies from 1.5 to 0.97 eV, depending on the substrate, but its position relative to the THAP HOMO-1 remains fixed (Fig. 2). In a separate experiment, CuPc directly deposited on high WF PEDOT:PSS (5.65 eV) gives up some charge to the substrate, which leads to a WF decrease similar to that observed for CuPc on the high WF THAP; yet, no new state is observed in this case. These observations suggest, therefore, that the interface state is linked to the THAP molecule, and corresponds to the occupation of a previously unoccupied THAP level, relaxed below E_F in the presence of the donor CuPc molecule. Recent observations of CuPc gap states resulting from the occupation of the molecular LUMO by an electron donated by an alkali metal donor[19] are consistent with our data. The gap state is clearly visible at the CuPc-on-THAP interface but not at the THAP-on-CuPc counterpart. A possible explanation for this asymmetry is as follows. At the CuPc-on-THAP interface, charge is transferred from an ultrathin CuPc overlayer (2–4 Å) to a state that overlaps the energy gap of the underlayer. The gap state is, therefore, clearly visible through the CuPc layer over the low density background of the THAP gap. At the other interface, the interface state is located in the ultrathin THAP layer, giving a low level signal that overlaps with the high density of state valence features of the CuPc underlayer.

An interesting situation emerges from the OOH energetic data presented above. Previous investigations of organic heterojunctions have led to the suggestion that interface dipoles between two organics can be semiquantitatively predicted based on the difference between the energy positions of the CNLs of the two constituent semiconductors.[4] In the IDIS model, the CNL can be understood as representing the "Fermi level" or the "chemical potential" of the intrinsic organic materials. Therefore, the initial offset between the CNLs gives rise to a charge transfer across the interface, which induces an interface dipole and tends to align the CNLs. This charge exchange is further screened by the dielectric constants of the materials.[4] A significant pool of data supports the model,[20] including data from OOH with one of the two organic films chemically doped. In the present case, however, the interfaces are formed between two relatively narrow gap materials (1.75 eV for THAP and 1.9 eV for CuPc), and the substrates are chosen so as to move the Fermi level of the system very close to, or beyond, the limits of these gaps. As E_F moves close to the HOMO or LUMO of these materials, in a region of the gap where the density of states becomes significant, a considerable amount of charge is exchanged, over and beyond the exchange caused by the simple alignment of the CNLs. The OOH energetics become, therefore, substrate dependent, as is demonstrated above.

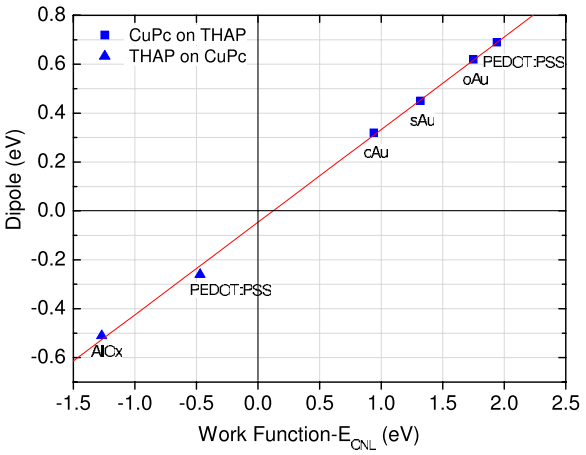

FIG. 5. (Color online) Linear fit of the interface dipole △ versus the difference between the work function of the underlayer and the binding energy of the charge neutrality level of the overlayer. ■ CuPc on THAP; ▲ THAP on CuPc.

Interestingly, for the pair of small molecules investigated here, we find a linear dependence of the interface dipole Δ on the starting work function of the organic underlayer, (Figs. 4 and 5). In Fig. 5, the scale on the positive horizontal axis corresponds to the difference between the initial THAP WF and the energy position of the CuPc CNL, i.e., 4 eV below the vacuum level,[4] and the scale on the negative horizontal axis to the difference between the initial CuPc WF and the position of the THAP CNL, i.e., 5 eV below vacuum level. Note that the THAP CNL is not calculated, but deduced from previous investigations of THAP on various substrates.[21] Very good linearity with a slope of 0.37 is obtained on both parts of the plot, and the linear fit goes through the origin within experiment error.

These results show that the organic overlayer "senses" the substrate WF through the thin (~50 Å) organic underlayer. As a result, its molecular levels follow the substrate Fermi level, as it would at an organic-metal interface. This was already clear from the observation that the molecular levels of the overlayer moves so as to always keep the HOMO or LUMO below or above E_F, respectively (Fig. 3). However, a more subtle result emerges from the linear dependence of Δ with a slope of 0.37: this slope is significantly larger than would be expected for an interface between a metal and an organic semiconductor with a gap of 1.7–1.8 eV. Slopes of 0.1–0.25 have been measured and calculated for materials including PTCDA[17] and CuPc.[22] On the other hand, the fact that Δ varies at all as a function of substrate shows that the molecular offsets at the organic heterojunction are not simply defined by the organic pair. With the very thin organic underlayer predeposited on the substrate, the electronic structure of the OOH depends on the substrate-underlayer interface, i.e., the Fermi level position and WF of the organic underlayer. The adaptability of molecular levels of the overlayer to the Fermi level of the system is mitigated by organic-organic interactions that are weak compared to those at the metal-organic interface. This result is presumably very

general, and has direct implications for systems where organic-metal interfaces are modified by an intermediate organic interlayer.

SUMMARY

The work described in this paper shows in a systematic way that the electronic structure of an organic-organic heterojunction, i.e., the molecular level offsets and dipole across the OO interface, is significantly influenced by the substrate, at least when the organic underlayer is thin (of the order of 5 nm or less). In that case, the position of the molecular levels of the overlayer and the OOH electronic structure respond to changes in the substrate work function in a way that is reminiscent of an organic/metal interface, however, mitigated by the presence of an intermediate organic film. We expect these results to shed light on the electronic structure of OOHs and interfaces between organic semiconductors and metal surfaces modified by intermediate layers.

ACKNOWLEDGMENTS

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